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(पहला पुनरीक्षण)

Indian Standard

CARBON BLACK, SUPER ABRASION FURNACE [SAF (N-110)] — SPECIFICATION

(First Revision)

UDC 661·666·4 : 678·4·046·2

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

May 1991

Price Group 3

Rubber Products Sectional Committee, PCD 13

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Carbon black is added to rubber to develop physical strength properties and is, therefore, commonly known as reinforcing agent.

This standard covers the requirement of super abrasion furnace carbon black used by the rubber industry and which has been given the nomenclature of N-110 by the American Society for Testing and Materials (ASTM).

This standard was first published in 1982. In this revision, Industry Reference Black (IRB) No. 6 has been included as reference black instead of IRB No. 4 for measuring physical properties of the vulcanizate. Also, the requirement for loss on heating has been modified.

This standard contains clauses **4.2** and **4.4** which call for agreement between the purchaser and the supplier.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

AMENDMENT NO. 1 APRIL 2003
TO
IS 10358 : 1991 CARBON BLACK, SUPER ABRASION FURNACE [SAF N-110)] — SPECIFICATION

(First Revision)

(*Page* 5, *clause* C-1, *line* 6) — Substitute 'IRB No. 7' *for* 'Indian Reference Black No 6'

(*Page* 6, *clause* C-4, *line* 1) — Substitute '30 minutes' *for* '15 and 30 min'.

(*Page* 6, *clause* C-6.1, *line* 3) — Substitute 'IRB No 7' *for* 'IRB No 6'.

(*Page* 6, *Table* 2) — Substitute the following for the existing:

Table 2 Difference in Physical Properties of Valcanizates Containing [SAF (N-110)] Carbon Black from IRB No. 7

Sl No.	Cure Conditions	Tensile Strength *Min*, MPa*	300 Percent Modulus MPa*
(1)	(2)	(3)	(4)
i)	30 min at 145°C	-2 66	-4 65 to -1 65

*1 MPa = approximately 10 2 kgf/cm²

(PCD 13)

Indian Standard

CARBON BLACK, SUPER ABRASION FURNACE [SAF (N-110)] — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the method of sampling and test for super abrasion furnace [SAF (N-110)] carbon black for use in rubber industry.

2 REFERENCES

The Indian Standards given in Annex A are necessary adjuncts to this standard.

3 TERMINOLOGY

For the purpose of this standard definitions given in IS 7503 (Parts 1 to 4) shall apply.

4 REQUIREMENTS

4.1 The material shall be free from foreign matter like wood, metal and fibres.

4.2 Pelletization

The material shall be delivered in the pelletized form. Pellet hardness shall be controlled to such a degree that satisfactory dispersion is obtained when compounded in standard mixing equipment as desired by the purchaser.

4.3 The material shall also comply with the requirements given in Table 1.

4.4 Compounding

If desired by the purchaser the material may be compounded in natural rubber test recipe following the procedure given in Annex C and the properties of carbon black assessed relative to IRB No. 6.

5 PACKING AND MARKING

5.1 Packing

The material shall be supplied in bags. The net mass of each bag shall be 25±0.5 kg. The bags shall be shaped to facilitate stacking in pellets by slight ironing.

5.2 Marking

The packages shall be marked with the name of the manufacturer, net mass, month and year of manufacture, batch number, grade identification [SAF (N-110)], and shall have violet colour as a colour code identification.

Table 1 Requirements for Super Abrasion Furnace [SAF (N-110)] — Carbon Black

(*Clauses* 4.3 *and* 6.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex	Cl No. in IS 7498 : 1985
(1)	(2)	(3)	(4)	(5)
i)	Iodine adsorption, as mg of iodine/g* of carbon black	143 to 158	—	5
ii)	Dibutyl phthalate (DBP) absorption, *ml*/100 g, of carbon black	108 to 118	—	6
iii)	Pour density, g/l	316 to 380	—	7
iv)	Sieve residue, percent by mass, *Max*			
	a) Through 45-micron IS Sieve	0.1000	—	8
	b) Through 500-micron IS Sieve	0.0010	—	8
v)	Loss on heating, percent by mass, *Max*	3.0	—	9
vi)	Ash content, percent by mass, *Max*	0.75	—	10
vii)	Fines content, percent by mass, *Max*	15	—	13
viii)	Discolouration of toluene, percent transmission, *Min*	80	—	16
ix)	Tinting strength	121±5	Annex B	—

*Iodine adsorption using 0.25 g carbon black

5.2.1 The packages may also be marked with the Standard Mark.

6 SAMPLING, NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

6.1 Sampling

The samples of carbon black shall be drawn in accordance with **2** of IS 7498 : 1985.

6.2 Number of Tests and Criteria for Conformity

All the characteristics of [SAF (N-110)] carbon black given in Table 1 shall be tested on individual samples. The lot shall be declared as conforming to the requirements of the specification, if all the test results of each of the individual samples satisfy the corresponding requirements.

ANNEX A

(*Clause* 2)

LIST OF REFERRED INDIAN STANDARDS

IS No.	*Title*
1675 : 1971	Stearic acid, technical (*first revision*)
3399 : 1973	Zinc oxide for rubber industry (*first revision*)
3400 (Part 1) : 1987	Methods of test for vulcanized rubbers: Part 9 Tensile stress-strain properties (*second revision*)
4588 : 1986	Rubber, raw natural (*third revision*)
7498 : 1985	Methods of sampling and test for carbon blacks (*first revision*)
7503 : 1985	Glossary of terms used in rubber industry.
(Part 1) : 1988	Definitions of basic terms (*first revision*)
(Part 2) : 1988	Definitions of additives (*first revision*)
(Part 3) : 1988	Definitions relating to properties and testing (*first revision*)
(Part 4) : 1988	Definitions relating to processing (*first revision*)
8483 : 1989	Dibenzothiazyl disulphide (*first revision*)
8851 : 1978	Sulphur for rubber industry

ANNEX B

[*Table* 1, *Sl No.* (ix)]

DETERMINATION OF TINTING STRENGTH

B-1 SCOPE AND FIELD OF APPLICATION

B-1.1 This specifies a method for the determination of the tinting strength of carbon black to an industry tint reference black.

The method is based on the use of two different but very widely used commercial instruments. Other instruments may be used but they shall have at least equivalent performance.

B-2 PRINCIPAL

Mixing of a test sample with zinc oxide and a liquid plasticizer. Mulling and homogenization of the paste obtained, then spreading as a layer of regular thickness Measurement of its light reflectance with a reflectometer sensitive to different shades of grey.

Mixing and measurement of the light reflectance of an industry tint reference black in the same manner and, from the two light reflectance values, determination of the relative tinting strength of the sample.

B-3 REAGENTS

B-3.1 Plasticizer: Soyabean Oil Epoxide — 0·92 to 0·99.

B-3.2 Industry Tint Reference Black

B-3.3 Zinc Oxide — conforming to IS 3399 : 1973.

B-4 APPARATUS

B-4.1 Analytical Balance — accurate to 0·1 mg.

B-4.2 Automatic Muller

B-4.3 Oven — capable of being maintained at 105 ± 2°C

B-4.4 Syringe — Automatic filling, accurate to 0·03 ml

B-4.5 Pelette Knives — Preferably tapered, of stainless steel, 100 to 150 mm long

B-4.6 Paste Application Apparatus

One of the following groups shall be used

B-4.6.1 *Film Drawdown Method*

B-4.6.1.1 *Film applicator* — 0·08 mm gauge depth, to give 0·04 mm wet film thickness.

B-4.6.1.2 *Glass plate* — approximately 750 mm × 500 mm × 10 mm.

B-4.6.2 *Roller Spreader Method*

B-4.6.2.1 *Tint roller spreader*

B-4.6.3 *Meeco Colormaster Method*

B-4.6.3.1 *Glass slides* — dimensions 75 mm × 50 mm × 1·22 mm ± 0·05 mm.

B-4.6.3.2 *Glass rod* — approximately 6·5 mm × 250 mm.

B-4.6.3.3 *Slide preparation apparatus* — (*see* Fig. 1).

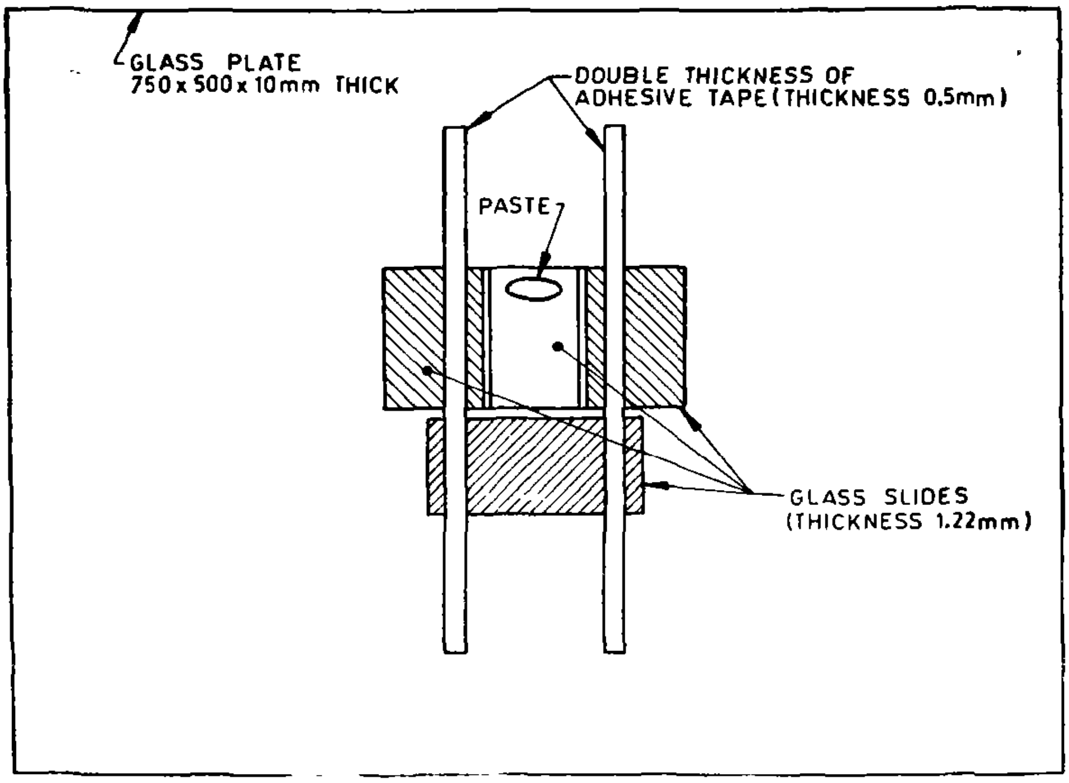

FIG 1 SLIDE PREPARATION APPARATUS

B-4.7 Light Measuring Instrument — sensitive to variations in light reflectance of shades of grey.

NOTE — The instrument shall be operated in accordance with the manufacturer's instructions for optimum performance.

B-5 TEST CONDITIONS

The test should preferably be carried out under standard temperature and humidity conditions as per standard laboratory conditions, that is, at 27±2°C and 65±5 percent relative humidity.

B-6 PROCEDURE

B-6.1 Dry the zinc oxide and carbon black in accordance with IS 7498 : 1985. Just sufficient industry tint reference black should be dried, as re-drying on a future occasion is not recommended.

B-6.2 Weigh 0·100 0 g of N 3 series and smaller particle carbon blacks or 0·200 0 g of other carbon blacks. (Crushing of pelleted carbon blacks may be beneficial). Weigh 3·750 0 g of the zinc oxide.

B-6.3 Using the syringe, place 2·2 ml of the plasticizer in the centre of the glass plate.

Place the zinc oxide and carbon black in the centre of the pool of plasticizer.

Mix the three materials thoroughly with a pelette knife.

B-6.4 Set the muller to a pressure of 0·017 MPa between the plates (445 N force when using 18·4 cm diameter plates), by placing the extra masses supplied with the equipment, on the arm. Close the plates and mull for 25 rev.

Open the plates, scrape the upper plate with a pelette knife to remove as much as possible and transfer it to the lower plate. Then, with the lower plate rotating, use the pelette knife to spread the paste to a flattened circle on the plate, and work all the paste to the centre. Repeat this step twice more.

Repeat this procedure three times, that is, for a total of four × 25 rev.

Paste should not be kept for more than 24 h.

B-6.5 Prepare pastes of the test sample and the industry tint reference black for light reflectance measurements by one of the following methods.

B-6.5.1 *Film Drawdown Method*

Clean the glass plate with a tissue to remove any dust particles or film. Using a clean palette knife, place a portion of the mulled paste at the top edge of the glass plate and smear it almost to the bottom edge. Using the film applicator, draw down the paste to the bottom edge in about 2 to 3 s.

B-6.5.2 *Roller Spreader Method*

Using a clean pelette knife, place a portion of the mulled paste on the clean turning roller of the tint roller spreader.

B-6.5.3 *Meeco-Colormaster*

Clean the glass slide with a tissue to remove any dust particles or film. Using a clean pelette knife, place a small portion of the mulled paste along the edge of the slide. Place the slide in the slide preparation plate (*see* Fig. 1) with the paste at the open end. Draw the glass rod across the sample from the open end towards the closed end three times, uniformly spreading the paste on to the slide. It is not necessary to cover the full length of the slide. The glass rod resting on the two slide strips of making tape gives a film thickness of 0·5 mm (*see* Fig. 1).

B-6.6 Set up and check the reflectance instrument in accordance with the manufacturer's instructions. Any external meter used shall be calibrated to give the same readings as the main instrument.

B-6.6.1 *Measurements Using the Densichron Reflectometer*

B-6.6.1.1 *Film drawdown method*

Set the range switch to 2 and operate with no filter in the reflection head. Place the reflection head aperture over a drawdown of the paste prepared from the industry tint reference black. Adjust the meter to read 80 percent for at least three readings, all taken 75 mm or more from the top of the drawdown.

NOTES

1 If results are variable, gather up the paste and make another drawdown.

2 Use the 4·762 5 mm (3/16 in) aperture if reflection head No. 3882 A is used.

Without altering the setting, take three readings from a paste drawdown prepared from the test sample (*see* Note 1 above).

B-6.6.1.2 *Roller spreader method*

With the film applicator in place, place a portion of the industry tint reference black paste on the turning roller. Turn the range switch from position 0 to position 2. Using the calibration control, adjust the meter to read 80 percent. Turn the range switch to position 0. Clean the roller and applicator. Place a portion of the test sample on the turning roller and turn the range switch to position 2. Record the reflectance value.

B-6.6.2 *Measurement Using the Meeco Colormaster*

When using the instrument, use the green filter only.

Place a slide prepared from the industry tint reference black with the paste side up centrally over the left front hole and immediately read the reflectance. Read the reflectance from slide similarly prepared from the test sample.

B-7 CALIBRATION OF THE APPARATUS

B-7.1 Prepare five standard pastes in duplicate, as described in **B-6.1** to **B-6.5** but using the masses of the industry tint reference black (ITRB) specified below (*see* **B-6.2**):

ITRB	*Calibration Tint Value*
g	percent
0·090 0	90
0·100 0	100
0·110 0	110
0·120 0	120
0·130 0	130

B-7.2 Prepare one of the pastes having a calibration tint value of 100 percent for measurement by the appropriate method specified in **B-6.5**.

B-7.3 Set up and check the reflectance instrument in accordance with the manufacturer's instructions. Any external meter used shall be calibrated to give the same readings as the main instrument.

B-7.3.1 *Densichron Reflectometer*

B-7.3.1.1 *Film drawdown reflectometer*

Set the range switch to 2 and operate with no filter in the reflection head. Place the reflection head aperture over a drawdown of the paste having the 100 percent calibration tint value. Adjust the meter to read 80 percent for at least three readings, all taken 75 mm or more from the top of the drawdown.

NOTES

1 If the results are variable, gather up the paste and make another drawdown.

2 Use the 4·762 5 mm (3/16 in) aperture if reflection head No. 3882 A is used.

Without altering the setting, take three readings from the duplicate paste having the 100 percent calibration tint value.

These pastes are acceptable if readings from 79·5 to 80·5 are obtained.

3 It is beneficial to have the two drawdowns side by side.

B-7.3.1.2 *Roller spreader method*

With the film applicator in place, place a portion of one of the pastes having the 100 percent calibration tint value on the turning roller. Turn the range switch from position 0 to 2. Using the calibration control, adjust the meter to read 80 percent. Turn the range switch to position 0. Clean the roller and applicator. Place a portion of the other paste having the 100 percent calibration tint value on the turning roller and turn the range switch to position 2. The pastes are acceptable if readings from 79·5 to 80·5 are obtained.

B-7.3.2 *Meeco Colormaster*

When using this instrument, use the green filter only. Place a slide prepared from one of the industry tint reference blacks having the 100 percent calibration tint value with the paste side up centrally over the left front hole and immediately read the reflectance. Read the reflectance from a slide prepared similarly from the duplicate industry tint reference black having 100 percent calibration tint value. If the reflectance is within ±0·6 percent, the two pastes are acceptable.

B-7.4 If the duplicate 0·100 0 g pastes are acceptable, blend the two together using a pallette knife.

B-7.5 If the duplicate 0·100 0 g pastes are not acceptable, prepare another 1·000 g paste and check by the appropriate procedure. Blend it with whichever of the previous pastes ensures compliance with the specified limits.

B-7.6 Following the appropriate procedure specified in clause **B-6**, determine the reflectance value of all the remaining ITRB pastes.

B-7.7 Calculate the average tinting strength for the duplicate determinations of the standard pastes ($R = 80$ if the Densichron reflectometer used).

B-7.8 A properly calibrated instrument will give average tinting strength values within ±0·8, relative to the calibration tint values given in **B-7.1**.

B-7.9 When an instrument is checked in accordance with the manufacturer's instructions and is found to be functioning correctly, but is found to give tinting strength values outside ±0·8 of the actual values for the standard pastes (**B-7.1**), calculate the regression of the standard values on the measured values by the method of least squares. Alternatively, plot a graph of observed values *versus* standard values.

B-7.10 Correct the test values of all subsequent samples by substituting each measured value in the equation and calculating the corrected value, or apply a correction factor from the graph.

B-7.11 Periodically check the proper calibration and standardization of the equipment, reagents, materials and method, as required, using standard reference blacks (**B-3.4**).

B-8 EXPRESSION OF RESULTS

The relative tinting strength, T, of the sample, expressed as a percentage, is given by the formula:

$$T = \frac{R}{S} \times 100$$

where

R = reflectance of the industry tint reference black, and

S = reflectance of the test sample.

If 0·2 g of test sample is used, divide the result by 2.

B-9 TEST REPORT

The test report shall include the following information:

a) Reference of this Indian Standard,

b) Identification of the sample,

c) Identification of the primary reference black,

d) The method of preparing the paste for measurement,

e) The reflectance instrument used,

f) The result of replicate tests, and

g) Any deviation from the procedures specified in this Indian Standard.

ANNEX C

(*Clause* 4.4)

SCHEDULE FOR COMPOUNDING AND TESTING FOR PHYSICAL EVALUATION OF CARBON BLACK

C-1 GENERAL

These procedures involve the incorporation of the black to be tested in rubber along with the necessary auxiliary agents, to permit vulcanization, followed by testing. Along with each test, blank, a corresponding stock containing the Industry Reference Black No. 6 is included. The differences between the properties obtained on the reference black is simply a device to cancel the inevitable variations in test results which are due to minor variations between laboratories in equipment, materials, procedures and ambient conditions.

C-2 STANDARD NATURAL RUBBER COMPOUNDING FORMULATION

The standard formulation for testing carbon black is given below:

Material	*Parts by Mass*
Natural rubber grade ISNR: 5 (*see* IS 4588 : 1986)	100
Zinc oxide (*see* IS 3399 : 1973)	5
Stearic acid (*see* IS 1675 : 1971)	3
Dibenzothiazyl disulphide (*see* IS 8483 : 1976)	0·6
Sulphur (*see* IS 8851 : 1978)	2·5
Carbon black [SAF (N-110)]	50

C-3 MIXING METHOD

C-3.1 The mixing method is given in **C-3.1.1** to **C-3.1.10**.

C-3.1.1 Use a two roll laboratory mill having 150 mm outside diameter and 250 to 280 mm working distance between the guides. The speed of slow roll should be 24±0·5 revolutions/minute and the friction ratio should be 1 to 1·4. Adjust and maintain roll temperature at 70±5°C and set mill opening at 1·4 mm.

C-3.1.2 The carbon black shall be conditioned before weighing, by heating in an oven at 100 to 110°C for one hour.

C-3.1.3 Weigh the ingredients for a batch size which is 4 times of the parts by mass, in g, given in **C-2**.

C-3.1.4 Add rubber and band on the mill, make two 3/4 cut from each side (time 2·0 minutes).

C-3.1.5 Set mill opening at 1·65 mm and add stearic acid and make 3/4th cut, once each way (*time 2·5 minutes*).

C-3.1.6 Add sulphur, accelerator, zinc oxide and make 3/4th cut, twice each way (time 2 minutes).

C-3.1.7 Add carbon black. Open mill gradually to maintain constant bank. Make 3/4th cut, three times each way after all carbon black is in (time 7·5 minutes).

C-3.1.8 Cut stock, roll and weigh. If the mass of the mixed batch is beyond the tolerance of ±0·6 percent, reject the batch (time 1 minute).

C-3.1.9 Pass end-wise, six times, at 0·8 mm opening, and sheet off at 2·2 mm finished gauge (time 2·5 minutes).

C-3.1.10 Condition the stock, for 1 to 24 h, at 27±2°C and cut out suitable slabs for vulcanization.

C-4 VULCANIZATION

The test pieces are vulcanized for 15 and 30 min at 145°C in a standard 4-cavity mould which gives sheets of dimensions 150 mm × 150 mm × 2 mm. The curing press shall be capable of exerting a minimum pressure of 3·5 MN/m^2 (approx 35 kgf/cm^2) on the cavity areas of the mould during vulcanization. After vulcanization the sheets shall be cooled immediately in water. Condition the vulcanized test slab for *16 to 72 h at 27±2°C before testing.*

C-5 TESTING

C-5.1 The vulcanized sheets are tested for 300 percent modulus, and tensile strength in accordance with IS 3400 (Part 1) : 1987.

C-6 PHYSICAL PROPERTIES

C-6.1 The maximum difference in physical properties of vulcanizates containing [SAF (N-110)] carbon black as compared to IRB No. 6 shall be as given in Table 2.

Table 2 *Difference in Physical Properties of* Vulcanizates Containing [SAF (N-110)] Carbon Black from IRB No. 6

Sl No	Cured Conditions	Tensile Strength, *Min* MPa	300 Percent Modulus, MPa
(1)	(2)	(3)	(4)
i)	15 min at 145°C	−2·41	−2·00 to +1·00
ii)	30 min at 145°C	−1·16	−1·50 to +1·50

Standard Mark

The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

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Doc : No. PCD 13 (1013)

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Printed at Swatantra Bharat Press, Delhi, India